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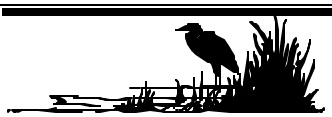

SCDHEC Environmental Laboratory Certification

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December 1998

Performance Evaluation Studies - Labs Located in South Carolina Only

Water Supply (WS) 041 Participants

The results for laboratories which participated in WS041 were mailed from this Office on October 21, 1998. Any laboratory which should have received results but did not should contact this Office immediately.

PE Studies Program Update - Privatization

The news of the privatization of the PE studies programs is that there is no Anew@ news. We=re still waiting for information from the USEPA and NIST. Our Office will notify all participating laboratories of instructions as soon as we receive them.

Turbidity -Hach Method 8195 Using StablCal Standards Accepted

The US EPA has determined that Hach Method 8195 using StablCal standards is an acceptable version of the approved EPA method 180.1 and can be used for NPDES and NPDWR compliance monitoring. For more information about this method, contact the Hach Company at (800)227-4224.

Environmental Management for Small Laboratories - Guide Available from EPA

In July 1998, the US EPA published the AEnvironmental Management Guide for Small Laboratories@. A small laboratory is Aone that has no full-time position dedicated to environmental management.@ The guide contains a summary of small lab activities and the federal environmental regulation that typically affect these activities, such as lab waste management, air quality management, wastewater management, a set of questionnaires that small labs can use to assess their relative environmental status, and a directory of applicable resources which included a listing of books, newsletters, meetings/conferences, state and federal contacts, and internet sites. The guide is available from:

Small Business Ombudsman
USEPA - Office of Policy Planning and Evaluation (2131)
401 M. Street
Washington, D.C. 20460.
Telephone number: (202)260-0490

Ask for document **EPA 233-B-98-001**.

A laboratory presents a unique environmental compliance and pollution prevention situation that can be very different from manufacturing and services oriented industrial processes. The purpose of the guide is to assist in the development and implementation of environmental management programs for small laboratories that meet important **Federal** regulatory requirements. It is imperative for small laboratory programs to understand that to be fully

responsive, the information provided by the guide must be supplemented by knowledge of applicable state and local regulations. The guide is designed to be a good starting source of information.

Microbiology Updates

New Information and Old Information Revisited

The *Manual for the Certification of Laboratories Analyzing Drinking Water*, Fourth Edition, Chapter V contains new requirements for laboratories certified for microbiological parameters. Lab Certification Officers have been informing laboratories of these new requirements during scheduled inspections. These new requirements are outlined below. Some old requirements, which are often found to be deficient, are also included. Laboratories must maintain documentation that shows the requirements have been met. This list is not all inclusive and must not be used as the sole source of information to evaluate compliance with method requirements.

It is strongly recommended that all laboratories obtain their own copy of the *Manual for the Certification of Laboratories Analyzing Drinking Water,* Fourth Edition (EPA 815-B-97-001) by calling the EPA Publications Unit at (800)490-9198. Currently, there is **no charge** for this document.

Hydrogen-Ion Concentration (pH)

If the manufacturer does not assign an expiration date to buffer solutions, the lab must assign an expiration date of one year after receipt.

If the pH meter is equipped with a temperature sensor or probe and it is used to measure temperature for compliance purposes, it must be checked annually with a certified thermometer and tagged with the date of the check, analyst=s initials and the correction value.

The electrode must be tagged with the in-service date.

Temperature Monitoring Devices

The mercury column in glass thermometers must not be separated. Inspect thermometers during the yearly calibration check.

Reference thermometers (NIST-traceable) must be recertified at least every 5 years. The thermometer must be checked at the ice point and a minimum of two other points that bracket the use of the thermometer.

If a thermometer differs by more than 1° C from the reference thermometer, it must be replaced.

It is strongly recommended that the thermometers used to check incubator temperatures be graduated in 0.1° C increments.

Incubators

An incubation temperature of 44.5° **"** 0.2°C can best be maintained with a water bath equipped with a gable cover and a pump or paddles to circulate water. Equipment must be upgraded if temperature fluctuations are noted.

Autoclave

A maximum temperature registering thermometer or a continuous recording device must be used during each autoclave cycle to ensure that the proper temperature was reached. The temperature recorded in the autoclave records must be read from the maximum temperature registering thermometer or the continuous recording device.

The automatic timing mechanism must be checked quarterly and the results recorded. This check should verify the time at sterilization temperature.

Autoclave door seals should be clean and free of caramelized media. Also, autoclave drain screens should be cleaned frequently and debris removed.

Microbiology Updates- continued

Hot Air Oven

If used for sterilization, a spore strip or ampule should be used on a monthly basis to ensure sterility of items.

Conductivity Meter

The cell constant must be determined monthly using a method indicated in Section 2510, AConductivity,@ in Standard Methods. Monthly calibration checks using an appropriate certified and traceable low-level standard may be substituted for determining the cell constant. The calibration must be within "10% of the true value.

If an in-line unit cannot be calibrated, it must not be used to check reagent-grade water.

Membrane Filtration Equipment

If graduation marks on clear glass or plastic funnels are used to measure sample volume, their accuracy must be checked with a standard graduated cylinder to verify an accuracy of #2.5%. If the mark is off by >2.5%, place a permanent mark at the proper level.

Filtration funnels must be completely wrapped in foil or char-resistant paper during sterilization and until used to filter samples.

Pipets

It is recommended that a hot air oven be used to sterilize pipets (if a hot air oven is available). Hot air oven sterilization alleviates the condensation that sometimes remains in the tips of the pipets after sterilization.

Pipets delivering volumes of 10ml or less must be accurate within a 2.5% tolerance. You may keep manufacturers specification sheet or test one pipet per lot by measuring the weight of the volume contained in the pipet. (1 ml of water weighs 1 gram).

Calibrated micropipetters may be used if tips are sterile. Micropipetters must be calibrated annually and replaced if the tolerance is greater than 2.5%.

Reagent Grade Water

If all other quality control tests are within limits, the bacteriological quality or toxicity test will not be required. Laboratories analyzing drinking water for total coliform bacteria using Colilert or Colisure are not required to perform quality control analyses on the reagent water used for the sterility check. The laboratory must use sterilized deionized or distilled water (not buffered dilution water) produced in-house or purchased from a commercial vendor. The water quality control tests are required if the laboratory is certified for additional microbiological methods.

Media

Each lot of commercially prepared medium and each batch of laboratory-prepared medium must be checked before use for performance with positive and negative control cultures.

Dehydrated media may now be kept until the manufacture=s expiration date, if it is not caked or discolored and as long as quality control tests are acceptable. The media must be kept in a cool, dry location. Desiccator storage is strongly recommended. Prepared A-1, LTB, BGBB, EC and PA liquid media may now be stored in the dark at <30°C. Prepared agar plates, m-endo and m-FC broth must still be refrigerated. HPC agar, stored in screw cap containers, may be kept for six months.

Laboratories are not required to check the pH of commercially prepared liquid media if they maintain the manufacturer=s pH certification. The pH certification must identify the lot number of the medium and must be available for review during the on-site evaluation.

Microbiology Updates- continued

Sterilization Procedures

Contaminated test materials must be autoclaved at 121°C for 30 minutes. Carbohydrate containing media must be autoclaved at 121°C for no longer than 12-15 minutes. Presence-absence broth must be autoclaved at 121°C for no longer than 12 minutes.

Analytical Methodology

Boiling water is no longer allowed to prevent bacterial carry-over between samples during a filtration series.

If UV light box is used for sanitation it must be tested quarterly with a UV light meter or agar spread plate. The lamp must be replaced if it emits less than 70% of its initial output or if an agar spread plate containing 200 to 250 microorganisms, exposed to UV light for two minutes, does not show a count reduction of 99%. These quality control checks of the UV light must be documented.

Alternatives to UV light sanitation to prevent bacterial carry over:

1) Use a sterile filtration funnel for each sample.
2) A blank can be filtered between each sample to check for bacterial carry-over.

If two or more analysts are available, each analyst must count the total coliform colonies on the same membrane monthly. Colony counts must agree within 10%.

When using membrane filtration techniques for total and fecal coliform determinations, each analyst involved with identifying colonies must perform a verification test at least once every three months. For laboratories having more than three analysts, multiple verification tests by different analysts will have to be performed in some months.

National Primary and Secondary Drinking Water Regulations:

Analytical Methods for Regulated Drinking Water Contaminants

Action: Direct Final Rule

On September 3, 1998, the EPA approved the use of updated versions of previously approved American Society for Testing and Materials (ASTM), Standard Methods for the Examination of Water and Wastewater (Standard Methods or SM) and Environmental Protection Agency (EPA) analytical methods for compliance determinations of chemical and microbiological contaminants in drinking water. At this same time the Agency is withdrawing approval of previous versions of the 14 EPA methods. Previous versions of the SM and ASTM methods will continue to be approved.

The Agency is promulgating these methods as a direct final rule because the Agency does not expect adverse comments and wants to ensure prompt availability of the methods for compliance monitoring. A direct final rule making involves publishing a rule with a delayed effective date as well as a companion proposed rule referencing the direct final rule and inviting public comment.

The Agency is also making minor technical corrections or clarifications to the regulations, amending the regulation to change the composition of Performance Evaluation (PE) samples and require successful analysis of PE samples once per year.

Effective Date: January 4, 1999, unless EPA receives relevant adverse comments by November 2, 1998.

ASTM and Standard Methods

These methods include 6 revised ASTM Methods and 23 revised Standard Methods. EPA is also approving the unchanged versions of 19 ASTM methods and 31 Standard Methods that are published in the 1996 ASTM and 1995 Standard Methods publications. EPA is not withdrawing approval of the currently approved version of any ASTM or Standard Method.

National Primary and Secondary Drinking Water Regulations - continued

Twelve of the revised Standard methods include eleven microbiology methods and one turbidity method. The eleven microbiology methods published in the 19th edition of Standard Methods have been extensively rewritten to improve clarity and ease of use. The revised methods contain certain steps that presently are only specified in the regulations at 40 CFR 141.21 (f).

EPA Methods

The fourteen revised EPA methods are contained in the manual AMethods for the Determination of Organic Compounds in Drinking Water, Supplement III@. The new versions contain minor corrections, minor technical enhancements and editorial improvements. The new versions also contain the mandatory method modifications published in the EPA document ATechnical Notes on Drinking Water Methods@.

New revision numbers for Methods 502.2, 504.1, 505, 506, 507, 508, 508.1, 515.1, 515.2, 524.2, 525.2, and 531.1 indicate a relatively minor modification to the method. New method numbers for Methods 551.1 and 552.2 indicate a relatively larger change in the method that significantly enhances the performance of the method. The differences in the methods are described in the Federal Register notice.

Annual PE Requirement

EPA amends the regulations to adopt the universal requirement for laboratories to successfully analyze a single PE sample at least once per year.

Sample Collection Procedures for Asbestos and Nitrate

The table at 40 CFR 141.23(k)(2) lists preservation procedures and holding times for several drinking water contaminants. EPA corrected the errors in this table related to measurements for asbestos, nitrate, and total nitrate (nitrate plus nitrite).

EPA is adding the 48 hour holding time and other instructions for collecting asbestos samples that were inadvertently omitted from the table. The preservation and holding times specified in EPA Method 100.2 apply to all compliance analyses of asbestos.

The regulations incorrectly list Anitrate@ as the analyte of concern in two types of sample: chlorinated drinking water and unchlorinated drinking water to which sulfuric acid has been added as a preservative. The correct analyte of concern in both cases is Atotal nitrate@. The preservative procedures and holding times for both entries are also incorrect. This rule combines the nitrate entries into one entry for total nitrate, adds an entry for nitrate-only determinations, and specifies correct preservation procedures and sample holding times for nitrate and total nitrate.

Nitrate and nitrite samples may be held up to 48 hours, if kept at 4^{o} C or less. These criteria are identical to those specified for wastewater samples at 40 CFR 136.3(e) and in EPA Method 300.0. Total nitrate samples may be held up to 28 days, if the sample is acidified. Drinking water samples may be held at ambient temperature, whereas wastewater samples must be held at 4^{o} C or less. A footnote is also added to this table to stress that if the sample has been acidified only total nitrate can be determined. Nitrate cannot be distinguished from nitrite in these samples because the acid and most disinfectants will oxidize nitrite to nitrate. If nitrate is to be measured separately from nitrite, the sample must not have been acidified or disinfected.

Analysis of Acid Herbicides

The herbicide 2,4-D is applied as an ester form and not as the acid 2,4-dichlorophenoxy acetic acid. The MCL for 2,4-D is listed in the table at 40 CFR 141.61(c) with the CAS number, 94-75-7. This CAS number is assigned to the ester, acid, and salt forms of 2,4-D. To clarify what is being regulated and analyzed, today=s rule changes the description of the contaminant in the table of approved methods at 40CFR 141.24(e) from A2,4-D@ to 2,4-D as acid, salts and esters@. This rule also ***National Primary and Secondary Drinking Water Regulations - continued***

clarifies in the footnotes to this table that Aaccurate determination of the Achlorinated esters@ of 2,4-

D and other regulated acid herbicides Arequires hydrolysis of the sample as described in Methods 515.1, 515.2, and 555". Laboratories need to ensure that they are performing the hydrolysis step as required by these methods.

SW-846 On-Line

The Office of Solid Waste (OSW) has made the electronic version of the SW-846 manual available on the internet. Access site http://www.epa.gov/oswer/test/main/htm. The on-line version contains all of the text, figures and tables found on the CD-ROM and printed versions of the manual, including all SW-846 methods through Update III and the proposed Update IVA. Files can be downloaded in PDF using the latest version of Adobe Acrobat Reader. OSW plans to update the SW-846 page quarterly, or as new method updates are available.. For more information, e-mail Gail Hansen at *hansen.gail@epamail.epa.com*.

Organic Updates

SW846 Revised to Incorporate Update III

On June 13, 1997, SW846 was revised to incorporate Update III. SW846 contains the analytical and test methods that EPA has evaluated and found to be among those acceptable for testing under subtitle C of the Resource Conservation and Recovery Act (RCRA). SW846 functions as a Federal guidance document setting forth acceptable, although not required, methods to be implemented by the user, as appropriate in responding to RCRA-related sampling and analysis requirements.

EPA has strongly recommended that the regulating entity develop a project-specific sampling and analysis plan in conjunction with laboratories and the regulating authority, to address both sample collection and method application and to assure the generation of data of the appropriate quality. To this end we have reviewed Update III and will address in this Update the South Carolina Certification requirements as they pertain to SW846 and other methods, to ensure the generation of quality analytical data from all laboratories certified by our Program.

Laboratories have already updated their standard operating procedures (SOPs) to incorporate the new SW846 requirements, therefore this information is reiterated to ensure that all laboratories will continue to meet the requirements as outlined below for any samples analyzed for reporting purposes to the Department. We are in the process of updating our certification requirements for organic analyses to incorporate Update III of SW846. If there are specific items you would like to see addressed please bring these to our attention.

Also, for several of the items discussed below, we are asking for your comments by a specific date. This does not mean that we are not open to comments on any of the other items. We are always open to comments and suggestions from any of the laboratories at anytime. Only by receiving feedback are we able to address your comments and improve our Program.

Method 8000B - Instrument Calibration

Method 8000B allows various options concerning initial instrument calibration and daily calibration verification. Some of the options allowed by this method and the GC/MS methods may affect the quality of the analytical data reported and therefore may not be considered as an acceptable option to the Department. This action is taken to ensure the quality of the analytical results reported by all laboratories. Each option will be expanded upon in the following text.

Organic Updates - continued

Non-linear Calibration Models

Non-linear calibration models (second and third order) are not to be used for those methods and/or instruments that have previously shown to exhibit linear calibration for the analytes of interest. For all of the methods and analytes certified to date, non-linear calibration has not been needed. There may be methods or analytes at some time in the future where a non-linear calibration is needed, but we have not encountered this need in the methods that we currently certify laboratories to perform.

In reviewing calibration data from organic laboratories to date, it has been noted that the laboratories using a non-linear fit had done so because of poor instrument response for one or more standards. This poor response is normally due to incorrect preparation of standard material, detector saturation at the high concentrations, or inadequate instrument sensitivity at the low concentrations. When instrument response is not adequate at the lower concentrations, this is validation that accurate quantitation at this concentration will be a problem. Maintenance can be performed to improve the response at this concentration or this standard can be dropped from the calibration data. As stated in Method 8000B, Section 7.5.3,@ it is not EPA=s intent to allow non-linear calibration to be used to compensate for detector saturation at higher concentrations or to avoid proper instrument maintenance. Thus, non-linear calibration must not be employed for those methods or instruments that have previously shown to exhibit linear calibration for the analytes of interest@.

Linear Calibration Using the Average Calibration or Response Factor

This calibration model assumes that the calibration curve is linear and passes through the origin when the relative standard deviation (RSD) is less than or equal to 20% for gas chromatography analyses (For GC/MS, <15% RSD for all compounds other than the calibration check compounds). The mean CF (calibration factor) or RF (response factor) and the standard deviation are used to calculate the %RSD. If any analytes exceed the acceptance limit for the % RSD, the following steps are to be taken:

1) Adjustments to the instrument operating conditions or other instrument maintenance may be needed. This is used where a linear instrument response meeting the 20% criteria has been met previously for this instrument.
2) Review the area counts, calibration or response factors, and RSD for those analytes to ensure that the problem is not associated with one of the five calibration standards. If the problem appears to be associated with a single standard, reanalyze that standard and recalculate the RSD. The standard solution may have been prepared improperly or a poor injection or purge may have resulted in poor response. The analyst=s experience will weigh heavily in determining what type of response is expected based on previous analyses. The analyst should review the response based on previous analyses to ensure consistency with previous data and take the necessary corrective actions.
3) The third alternative is to narrow the calibration range by replacing one or more calibration standards with standards that cover a narrower range. In most cases, it is the lower or higher standards that result in not meeting the method linearity requirements. Instrument sensitivity will affect an analyst=s ability to calibrate at a low concentration. Many analysts calibrate their instruments with six or more standards to enable them to drop the low standard and still have five standards for calibration. If linearity can be achieved using a narrower range, document the linearity and proceed with analyses.

The laboratory=s reporting limit is established by the concentration of the lowest standard analyzed during the initial calibration. Therefore, when narrowing the calibration range by changing the concentration of the lowest standard, the laboratory=s reporting limit will also be changed. It is very important that the reporting limit accurately reflect the instrumentation=s capabilities.

Organic Updates - continued

Linear Calibration using Linear Regression

After performing the steps outlined above, if the % RSD acceptance criteria cannot be met the analyst is allowed to use linear regression and document a correlation coefficient of at least 0.99 to verify calibration linearity. Many laboratories choose to use linear regression over the use of response factors or calibration factors. This is acceptable as long as the analyst goes through the three steps outlined above to verify acceptable responses for all analytes at all concentrations. When using linear regression the data system must document the calculated slope, y-intercept, and correlation coefficient. The data system must be able to print out the concentrations and responses for each analyte where a linear regression calculation is used.

Verification of Linear Calibrations

Verification of linear calibrations involves the calculation of % drift or % difference of the instrument response between the initial calibration and subsequent analysis of the verification standard. If using a linear regression equation, the % drift must be calculated, and if using an average calibration or response factor, the % difference must be calculated and documented for each analyte. If the percent drift or difference do not meet the method criteria, then no sample analyses are to be performed.

For Methods 8260B and 8270C, acceptance criteria for the percent drift or difference must be established for all the analytes of interest. This will ensure that all analytes are reviewed for adequate response for calibration verification before samples are analyzed. These methods currently only address the acceptance criteria for the calibration check compounds (CCCs), and EPA has stated that this will be corrected in the next revisions of these methods.

Unacceptable Options to SCDHEC - Method 8000B

Initial Calibration

Method 8000B also allows another option to analysts, where the RSD of an analyte may exceed the allowed 20% RSD for gas chromatography and 15% RSD for GC/MS analyses. This option allows an analyst to calculate the mean of the RSD values for all analytes in the calibration and if this value is less than or equal to 20% (15% RSD for GC/MS methods), then the calibration is deemed valid, but the analyst must provide the data user with the list of compounds for which the RSD exceeded the method criteria. Method 8000B documents that the analyst and data user must be aware that the use of this approach will lead to greater uncertainty for those analytes for which the RSD is greater than 20% (15% for GC/MS methods). Because the Department does not want data that are questionable, this option is unacceptable. We are certain that most laboratories are interested in providing high quality data to their clients and the Department and agree that this option will lead to equivocal data.

Calibration Verification

Method 8000B, also allows the analyst to average all the responses for all analytes and if the average response is less than 15%, the calibration has been verified. This option is also unacceptable to the Department because the quantitative results, for those analytes where the difference is greater than 15%, will include a greater level of uncertainty. We are certain that most laboratories are interested in providing high quality data to their clients and to the Department and agree that this option will lead to equivocal data.

Method Blank Analysis

The results for the method blank analysis must document that all target analytes are below the applicable laboratory=s reporting limit (lowest standard used for initial calibration) for initial calibration, calibration verification, and analyses. This requirement applies to all analytes, including methylene chloride and acetone which are common laboratory contaminants.

Organic Updates - continued

Laboratory Control Samples (LCS)

As required by EPA Method 8000B, the LCS must fall within the acceptable range of 70-130% recovery. Because the LCS is prepared in a clean matrix, the laboratory should have few problems in meeting this requirement for most compounds. Some compounds will have problems meeting this criteria, for instance the phenol compounds for EPA Method 8270C. For these compounds, the SOP must specifically address the steps the laboratory goes through to ensure the best possible performance. The LCS is used as a guide for the laboratory in determining if there are procedural or matrix effects; therefore, the LCS recovery must fall within 70-130% as much as possible. For that reason, limits of D (detection) to >150% are not acceptable for the LCS samples. Although statistical data can be helpful, the laboratory needs to develop in-house limits that reflect the typical expectations of the instrumentation or method employed. For instance, if the method generally performs within 80-120% recovery limits for the LCS for a given compound, but the statistical limits ("3s) result in limits of 20-180% recovery, the analyst should not accept an LCS documenting 20% recovery for this same compound. The analyst should be concerned when the recovery falls below 80% typical performance and begin corrective action around 70-75% recovery.

Analytical Standards

EPA Methods 8260B and 8021B both changed the holding time requirement for the standards. It is now stated that stock standards have a six month holding time, except for gases which have a one week holding time. This holding time begins when the seal on the standard is broken. This further increases the importance of documenting the opened date for each vial in the standard preparation records. For secondary standards, any standard prepared as a dilution of a stock standard, is assigned a one week holding time. This applies to all compounds, gas and non-gas. These holding times can be lengthened by verifying the standard. In order to verify the standard, the standard must be within 20% of the true value. This check must be performed each day that the standard is used beyond the holding time.

This Office has noted that when laboratories experience problems meeting the method required QC for these methods, the standard(s) are generally beyond the method defined holding time.

Confirmation Requirements for Gas Chromatography Analyses

When performing gas chromatography analyses, positive identifications for target analytes must always be confirmed using a second dissimilar analytical column or an alternate detector. This has always been a requirement for certification for the gas chromatography analyses and pertains to all analytical results submitted to the Department. It is acceptable to omit the confirmation procedure if the analyses are performed in conjunction with a remediation project and historical data is used to indicate the presence of specific contaminants. This must be thoroughly documented in the analytical results and the laboratory must have the historical data on file for verification.

Confirmation analyses must meet the same linearity and daily calibration criteria as for the primary analyses. Sample chromatograms from the confirmation column or detector must be evaluated using the same criteria as for the primary column. Although only those target analytes tentatively identified on the primary column need be evaluated, it is often useful to review the confirmation data as a separate analysis, for possible use as the primary analysis due to interferences and integration difficulties frequently encountered with complex sample matrices.

Reporting Limits for Analytical Results

The laboratory=s reporting limit for each analyte is established by the concentration of the lowest standard analyzed during the initial calibration procedure. When reporting analytical results, a reporting limit must be documented for each analyte and adjusted for any dilutions that have been performed on the sample.

Organic Updates - continued

Elevated Reporting Limits

It has been noted by SCDHEC Program areas that some of the results received for regulatory compliance data have elevated reporting limits that do not provide them with the information needed to determine compliance or noncompliance with environmental regulations. Many times laboratories are prevented from quantitating or identifying certain contaminants due to matrix interferences. This is understandable if the laboratory has taken all the necessary steps to eliminate or reduce the interferences, but these steps must be documented.

We are also aware that analytical laboratories may dilute a sample prior to analysis to avoid encountering interferences that may adversely affect their instrumentation. Laboratories can collect an extra sample for screening purposes to determine at what level the contaminants are present in the sample and if it actually needs to be diluted prior to analysis. The laboratory can set up different dilutions to analyze, starting with the most dilute sample and observing each dilution for interferences that may affect the quantitation of key analytes. Analytical results must indicate if dilutions are performed and document the interferences encountered in each dilution that prevented the laboratory from meeting the reporting limits needed by the Program area for specific analytes.

Reporting Limits for New Solid Waste Industrial Landfill Regulations

The Bureau of Land and Waste Management has requested that we address the reporting limits required for the new Solid Waste Industrial Landfill Regulations. It seems that laboratories have been characterizing the waste streams for disposal using reporting limits based on hazardous waste limitations. The reporting limits for the methods employed must be based on drinking water reporting limits. Reporting limits based on hazardous waste limitations can be as much as 100 times greater than the drinking water limits. All data submitted to the Department with inappropriate reporting limits will be rejected and the waste stream must be reanalyzed to obtain the required reporting limits. The Department cannot properly classify a landfill without the required reporting limits being met. Failure to meet the required reporting limits for the specified contaminants will result in a higher classification for the landfill. Questions should be directed to F. M. ABubba@ Carnes at (803)896-4121.

Volatile Organic Compounds (VOCs) - Methodology Changes

SW846 Update III changed the soil collection and analysis procedures for volatile organic compounds (VOCs). The updated procedures are contained in SW846 (Update III) Methods 5030B and 5035. The revised RCRA methods (SW846, Update III) require different sampling and analysis procedures for samples having high concentrations of VOCs versus low concentrations of VOCs. Update III sample collection techniques are more complicated and tedious for volatile organic analysis than those in the previous update; however, the accuracy of the Update III soil collection techniques warrant their use. Previous methodology has been shown to report significantly lower concentrations of VOCs in soil.

As of June 30, 1998, Method 5035 was required for the volatile analysis of all low concentration soil samples analyzed for reporting purposes to the State of South Carolina. The new low-level volatile analysis will require increased coordination between the field personnel and laboratory personnel. When collecting the samples, the appropriate sample containers, chain-of-custody forms, and sample collection instructions must be obtained from the laboratory performing the analysis prior to sampling for volatile organics.

Method 5030B incorporates the analysis of aqueous samples, soils, and other solid samples with a high VOC concentration (greater than 200 ìg/kg) or a high concentration oily waste sample (greater than 200 ìg/kg) using the conventional purge and trap apparatus. This procedure is used in conjunction with EPA Methods 8015B, 8021B, and 8260B.

Method 5035 describes the collection and analysis of low-level VOC solid samples (soils, sediment, and solid waste with VOC concentrations in the range of 0.5 to 200 ìg/kg). The analysis

consists of a closed system purge-and-trap method. Method 5035 utilizes a hermetically-sealed sample vial, the seal of which is never broken from the time of sampling to the time of analysis. Since the sample is never exposed to the atmosphere after sampling, the losses of VOCs during sample transport, handling, and analysis are negligible. The method also describes the procedures for collecting and preparing solid samples and oily wastes containing high concentrations of VOCs. This procedure is used in conjunction with EPA Methods 8015B, 8021B, and 8260B.

Method 5030B Ground-Water Sample Collection

Standard 40 mL glass screw-cap VOA vials with Teflon-lined silicone septa are to be used for collecting water samples for volatile analyses. Samples must always be collected in duplicate. The sample container needs to contain the necessary preservative, and the water should be introduced into the vials slowly without introducing any air bubbles within the vial. The vials should be completely filled at the time of sampling, so that when the septum cap is fitted and sealed, and the vial inverted, no headspace is visible. The vial must not be opened prior to analysis to preserve its integrity. Immediately after collection, the sample vials must be labeled and stored at $4^{o}C$.

Method 5035 Solid Sample Collection

There are various options that can be used when collecting soil samples for low-level VOCs. As with any sampling procedure for volatiles, care must be taken to minimize the disturbance of the sample in order to minimize the loss of volatile components. Always wear gloves whenever handling the tared sample vials. Several techniques may be used to perform the transfer of the sample to the relatively narrow opening of the low concentration soil vial such as the Encore™ sampler, a cut off disposable plastic syringe, or a stainless steel spatula.

The Encore™ sampler is both a sampler and a container for low-level soils. It is designed to collect an average weight with the exact weight to be determined in the lab. It is disposable and is also designed to have zero headspace. If the Encore™ sampler is used, the field personnel must get the sample to the laboratory within 48 hours of collection. A separate sample needs to be collected to enable the laboratory to perform a pretest on the soil to determine if the soil sample contains carbonate minerals that will effervesce upon contact with the acidic sodium bisulfate preservative solution in the low concentration sample vial. If the sample cannot be preserved with sodium bisulfate, the sample is then transferred to a preweighed vial with 5 mL of reagent water added to it. This sample in the unpreserved vial must either be analyzed immediately (within 48 hours of collection) or frozen within the 48 hour time frame and then analyzed within the 14 day holding time. Extra samples must still be collected for high concentration analysis, screening, and moisture content.

If using the disposable plastic syringe, collect several trial samples with the plastic syringe and weigh each trial sample, noting the length of the soil column in the syringe. Use the data to determine the length of soil in the syringe that corresponds to 5.0 g. Use one of the trial samples to determine whether or not the sample effervesces with the preservative. If so, collect the sample in a vial containing only lab pure water. Discard each trial sample. The sample is then collected and put into a preweighed vial, provided by the laboratory, with the sample preservative and a stir bar. A portable balance (capable of weighing to 0.01 g) can then be used to weigh the sealed vial containing the sample to ensure that approximately 5.0 g (3 - 7 g) of sample has been added to the vial. It is very important that the transfer of the sample be made as quickly as possible with very little disturbance to the soil to avoid loss of volatiles. The weight determination must be documented on the sample container and in the field records. The balance calibration needs to be verified in the field using an appropriate weight for the sample containers employed. The appropriate reference weight must be used at least once daily prior to weighing any samples and records must be kept for the balance checks.

The portable balance can also be used for weighing the preweighed vial and soil sample

collected

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using a stainless steel spatula. The weighing of the soil sample must be performed as quickly as possible to avoid the loss of volatiles. The preweighed vial must contain the preservative and a stir bar. Again, check the soil for effervescence prior to collection of the sample to be used for analysis.

All low-level soil samples must be collected in duplicate to allow the laboratory an additional sample for reanalysis. The second soil sample must be collected from the same soil stratum or the same section of solid waste being sampled and within close proximity to the location from which the original sample was collected. Additional samples must be collected for screening, dry weight determination, and high concentration analysis (if necessary) without the preservative. If high concentration samples are collected in vials containing methanol, an additional sample should be collected for screening and dry weight determination in a vial without preservative.

The laboratory performing the analysis needs to be contacted prior to sample collection to ensure that all necessary containers (with or without preservative) are available and that the proper sampling technique is used. Options for sample collection appear below:

Option 1 - Encore

1) Core with the Encore™ device in the field.
2) Collect two Encore™ samples/sample location*.
   *Shipment required day of collection.
3) Collect one glass container (2 oz.) with septum lid for the high-level (unpreserved*) and to determine moisture content.
   *Shipment required day of collection.
4) Collect one headspace vial for screening.
5) Laboratory preservation/preparation within 48 hours of collection for the Encore™ samples and high-level unpreserved bulk sample.

Option 2 - Low-level Vials

1) Core and weigh samples in field. (Balance required).
2) Collect two low-level vials (preserved or unpreserved*). Vials must be obtained from the laboratory performing the analysis.
   *Shipment required day of collection.
3) Collect one glass container (2 oz.) with septum lid for high-level (unpreserved*) and moisture determination.
   *Shipment required day of collection.
4) Collect one headspace vial for screening.
5) Laboratory preservation/preparation within 48 hours of collection for low-level unpreserved sample and high-level unpreserved bulk sample.

Option 3 - Field Screening

1) Field Screening.
2) Core and weigh in field.
3) Collect 2 low-level vials (preserved or unpreserved*)
   *Shipment required day of collection, or
   One high-level vial (unpreserved*).
   *Shipment required day of collection.
4) Laboratory preservation/preparation within 48 hours of collection for low-level unpreserved sample and high-level unpreserved bulk sample, or
   One high-level vial.

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Method 5035 Sample Preservation

Method 5035 addresses the preservation of the low concentration soil samples with sodium bisulfate to ensure a sample pH of # 2. If using option 2, two pre-weighed sample vials with the sodium bisulfate preservative solution must be obtained from the laboratory along with two pre-weighed sample vials with 5 mL of reagent grade water (used if vigorous effervescence). The laboratory will also provide a sample vial to check the reaction of the soil with the sodium bisulfate preservative solution. Soil samples that contain carbonate minerals (limestone) may effervesce upon contact with the acidic preservative solution in the low-level concentration sample vial. If the amount of gas generated is very small, any loss of volatiles as a result of such effervescence may be minimal, if the vial is sealed quickly. If at all possible the sample must be preserved. A test sample should be collected, added to a vial with the preservative and checked for effervescence. If a rapid or vigorous reaction occurs, discard the sample and collect low concentration samples in vials that do not contain the preservative solution, but contain 5 mL of reagent water. Soil samples with no preservative must be analyzed within 48 hours of collection or frozen. Records must be maintained on the chain-of-custody documenting the necessary actions taken by the laboratory.

Soil samples for volatile analysis that are preserved with the sodium bisulfate preservative solution are to be cooled to approximately 4^{o}C, packed in appropriate containers, and shipped to the laboratory on ice. These are to be analyzed within 14 days of sample collection. Samples receiving no preservation other than cooling to 4^{o}C must be shipped to the laboratory the same day that they are collected. Once in the laboratory, the samples must be analyzed or frozen within 48 hours of sample collection. The sample storage area must be free of organic solvent vapors. All samples need to be analyzed as soon as practical, and within the designated holding time from sample collection.

High Concentration Soil and/or Waste Samples

High Concentration soil and/or waste samples can be collected using the following methods: samples can be collected and preserved in the field in methanol, as a bulk sample in a VOA vial, or in an Encore™ sampling device. If a bulk sample is collected in a vial, the vial must be filled as completely as possible leaving no head space to avoid any loss of volatiles. It is imperative that the sample be received by the laboratory within 48 hours, so that the sample can be transferred to a vial with methanol preservative. Samples received in the Encore™ sampling device must be transferred within 48 hours of sample collection to a vial with methanol preservative. EPA Region IV discourages the use of methanol in the field due to transportation and contamination problems. If samples are collected in the field in methanol, a methanol field and trip blank must accompany the sample vial to verify any contamination from transportation or from the sampling site.

If methanol preservation in the field is used, the sample weight obtained in the laboratory will be used for the calculation of sample results. It is very important that the vial with methanol provided by the laboratory be returned to the laboratory as soon as possible after sample collection.

Method 5035 Certification

At the present time we have 27 laboratories certified to perform Method 5035. A laboratory=s certificate will document certification for Method 5035 or Method 5030, but it presently does not document the determinative method that applies to the specific sample preparation and extraction methods. A laboratory is only certified for Method 5035 in conjunction with the determinative method applied for during the application period. This is due to software changes needed to the Laboratory Certification Program=s database. When are in the process updating the software, but until this is completed to verify a laboratory=s certification, please contact this office at (803)935-7025.

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Traceability of Sample Containers for Method 5035 (Request for Comment)

To ensure that appropriate sample containers are used by the field personnel and that these containers are returned to the laboratory for sample analysis, we are proposing that the laboratory be required to maintain a log for the sample containers as they are issued to the sample collectors. Some laboratories have already established a system of traceability for sample containers.

The records for vials containing methanol must document a vial identification number, amount of methanol, weight to 0.01 g, date prepared, analyst=s initials, date relinquished/weight to 0.1 g, date returned to lab, weight of vial and sample, weight of sample, and analyst. The vial must be returned to the laboratory within 48 hours or the vial must be weighed in the field to ensure that there is no loss of methanol from the time the vial is prepared in the lab until the sample is collected in the field.

The records for the vials containing sodium bisulfate must document a vial identification number, amount of sodium bisulfate solution added to the vial, weight of the vial and preservative solution, date prepared, analyst=s initials, date relinquished/weight to 0.1 g, date returned to lab, weight of vial and sample, weight of sample, and analyst.

These records are to be maintained to ensure that the vials provided by the laboratory for sample collection are used before they can be contaminated. We are proposing this to laboratories, because of the cost of the vials and other sample containers for volatile organics. Your comments on this proposal are welcomed. **Please submit all comments by December 1, 1998 to the Office of Environmental Laboratory Certification.**

Sample Custody Issues with Method 5035 (Request for Comment)

It has come to the attention of this Office that samples collected in the Encore samplers are being transferred to a vial containing the sample preservative by an intermediate laboratory and then the sample is shipped on ice to the analytical laboratory for analysis. This procedure is followed in order to meet the 48 hour holding time required for the Encore sampling devices. This presents a problem with maintaining the sample integrity required for chain-of-custody purposes. EPA is being consulted on the issue of how to assure that the integrity of the sample will not be compromised.

When this transfer is performed in an intermediate laboratory the analytical laboratory cannot guarantee that the integrity of the sample will not be compromised. This is a decision that will have to be made by the analytical laboratory and if they have confidence in an intermediate laboratory performing this transfer. The analytical laboratory will need to ensure that the intermediate laboratory has a fresh supply of vials on hand and that they are kept in an area free of volatile contaminants. The transfer must also be performed in an area free of volatile organics. The equipment for performing weight measurements must be available in the intermediate laboratory and appropriate calibration records must be maintained. The analytical laboratory must also ensure that the transfer of these samples is correctly documented on the chain-of-custody form with the required preservation.

This problem will be eliminated if the holding time for the Encore samplers is extended, but at this time the EPA has not made the decision to extend this holding time. If the transfer is being made by an intermediate laboratory, documentation must be maintained on the chain-of-custody form to document the date and time of transfer along with the signature of the person who is performing the transfer. The intermediate lab must document the sample weight once the sample is transferred from the Encore into the vial with the sodium bisulfate preservative or a vial with methanol. This weight must be documented and transferred to the sample container. Appropriate quality control records must be kept for the balance used in making these weight determinations.

Once received by the analytical laboratory, the weight must be verified and documented. It is especially important to verify that there has been no loss of methanol for the high concentration samples.

We would like to receive comments on this issue from laboratories that have been performing these transfers as an intermediate laboratory and from the analytical laboratory concerning any

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problems they have encountered when this procedure is used. **Please submit comments on this issue by December 1, 1998 to the Office of Environmental Laboratory Certification.**

Notice to the Regulated Community

Embedded Microprocessors and the Year 2000

The South Carolina Department of Health and Environmental Control is actively working to assure all agency computer hardware and software systems will continue to function in the year 2000 and beyond. As part of that effort, the agency hereby advises the regulated community of potential problems with microprocessor-controlled equipment and devices used in the conduct of their business.

Information systems (hardware and software) used for essential business activities should be assessed for Year 2000 compliance, and, if necessary, renovated or replaced to achieve compliance. This includes devices, such as laboratory and communication equipment, which contain a microprocessor. It is possible these devices may not work properly after the year 2000, and could affect your compliance with state and federal regulations.

You are encouraged to contact the manufacturers of any such devices and obtain a Year 2000 Certification for the equipment. Many certifications are already posted on the companies= world-wide web sites.

Questions?

Q: How are the results reported for a BOD sample in which none of the dilutions deplete 2.0 mg/L?

A: The laboratory should use as many dilutions necessary so that at least one dilution will deplete at least 2.0 mg/L with 1.0 mg/L remaining. This could mean using a 100% dilution (300 ml of pure sample). If no dilutions meet this criterion, the demand should be calculated as a Aless than@ value based on the actual depletion of the dilution with the highest portion of sample. The depletion value should be multiplied by the reciprocal of the fraction of the sample used to make the dilution which has the most sample. For instance, if a sample were analyzed using 30, 60, and 150 ml in each of three dilutions and none of the dilutions depleted at least 2.0 mg/L, but the dilution with the most sample (150 ml) depleted 1.3 mg/L, the result would be reported as <2.6 mg/L. This answer is derived by multiplying 1.3 mg/L (the depletion value) by 2, the reciprocal of 150 ml (sample volume used) divided by 300 ml (total volume of dilution). If the highest dilution used only 60 ml of the sample and the depletion were 1.3 mg/L, the answer would be : 1.3 mg/L X (300 ml/60 ml) = < 6.5 mg/L. This is how results should be ***recorded*** on the laboratory bench sheet.

For information on how BOD results or the results of any other parameter tested for compliance with NPDES permits should be ***reported***, please contact Anthony James in the

Waste Water Compliance Assurance Section. His number is (803)734-5219.